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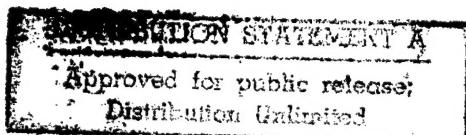
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THE PRODUCTION OF DEUTERIUM OXIDE AND THE DETERMINATION
OF ITS DENSITY

- USSR -

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THE PRODUCTION OF DEUTERIUM OXIDE AND THE DETERMINATION
OF ITS DENSITY*

This is a translation of an article written by A. I. Shatenshteyn, L. M. Yakimenko, V. R. Kalinachenko and Ye. A. Yakovleva, which was published in Zhurnal Neorganicheskoy Khimii (Journal of Inorganic Chemistry) Vol II, No 5, Moscow, 1957, pages 985-994.

The volume of a mixture of deuterium oxide and protium oxide is equal to the sum of the volumes of the components. This is a prerequisite for the derivation of the Longsworth-Swift equation [1, 2], which establishes the dependence of the density of water on the concentration of deuterium. To calculate the corresponding constants, it is necessary to know exactly the density of both deuterium oxide and protium oxide [3]. Many studies [4-12] have been devoted to the production of deuterium oxide and the determination of its density, as well as to the determination of its coefficient of thermal expansion.

The current study was undertaken in order to verify and increase the precision of the existing data. It is very important to compare the results of independent measurements of the density of deuterium oxide, which is of fundamental importance for the isotopic analysis of heavy water. It is for this reason that this paper is published although similar studies [13-15] have appeared in print after the completion of our work.

Twenty kilograms of heavy water ($d_{25}^{25} = 1.10752$, containing 99.81 at.-%D), to which 750 g of potassium tetroxide (K_2O_4) were added, were subjected to electrolysis to obtain pure deuterium oxide. The weight of the electrolyte at the end of the experiment, which lasted without interruption

* The work was completed in 1950 with the participation of G. V. Paletskiy, B. K. Danchenko, A. V. Zakrzhevskaya, V. M. Trokhova, R. A. Markozyan, Z. A. Tkachek, T. F. Mezhetsitskaya, A. I. Trekhunkova, V. I. Podolyan, A. Ya. Mamonina, A. A. Volkova and V. M. Dolinina.

for 15 days, was 1.8 kg; the concentration of deuterium, calculated according to Reley's [Rayleigh's ?] formula, reached 99.99997 at, %D.*

The apparatus described below enabled the following samples of water to be obtained:

- A -- deuterium oxide, synthesized from deuterium and electrolytic oxygen;
- B -- deuterium oxide, synthesized from deuterium and atmospheric oxygen;
- B¹-- deuterium oxide, synthesized by oxidation of deuterium over a cupric oxide which has the isotopic composition of atmospheric oxygen;
- C -- water, synthesized from compressed hydrogen and electrolytic oxygen.

The comparison of the density of samples H and B and the measuring of densities of sample C permitted us to judge the degree of enrichment of heavy water with the heavy oxygen isotope.

Gases subjected to synthesis were carefully purified and all measures were taken which guaranteed the conservation of the isotopic composition of atmospheric oxygen. Special methods permitted the purification of deuterium oxide, the measurement of its electrical conductivity, and the simultaneous filling of three pycnometers in the absence of air. The specific resistance of the purified deuterium oxide varied between 400,000 and 1,000,000 ohm. The density of samples was measured during the whole course of electrolysis. Two instruments were alternately used for the purification. Two sets of pycnometers, calibrated many times by two persons independently, were also utilized.

The basic measurements were carried out at 25°. The density of deuterium oxide was also measured at 30, 40, and 50°.

Description of the Equipment to Produce 100% Deuterium Oxide

Figure 1 is a diagram of the equipment. A one-celled electrolyzer $l(d = 30 \text{ cm})$, with electrodes of 700 cm surface area served for carrying out the electrolysis.

The electrodes were hollow and were cooled internally by a stream of water. The cathode was made of iron, and the anode of nickel-plated iron. An asbestos diaphragm separated the two electrodes. The electrodes were insulated from the

* The volume diminished seven-fold; $\alpha = 5$.

frame by a layer of paranite. The electrolyte entered the anode and cathode area of the cell from below; the gases were led upwards separately from the cathode and anode part of the cell through the opening into the frame. The electrolyzer was provided with a nozzle (2) to take electrolyte samples. Two supply tanks (5) with a capacity of 13 l each were used. Their presence guarantees the circulation of electrolyte and the equalization of the concentration of deuterium and alkali in the electrolyte. They are situated over the electrolyzer. Sight glasses (3) are located under the tanks.

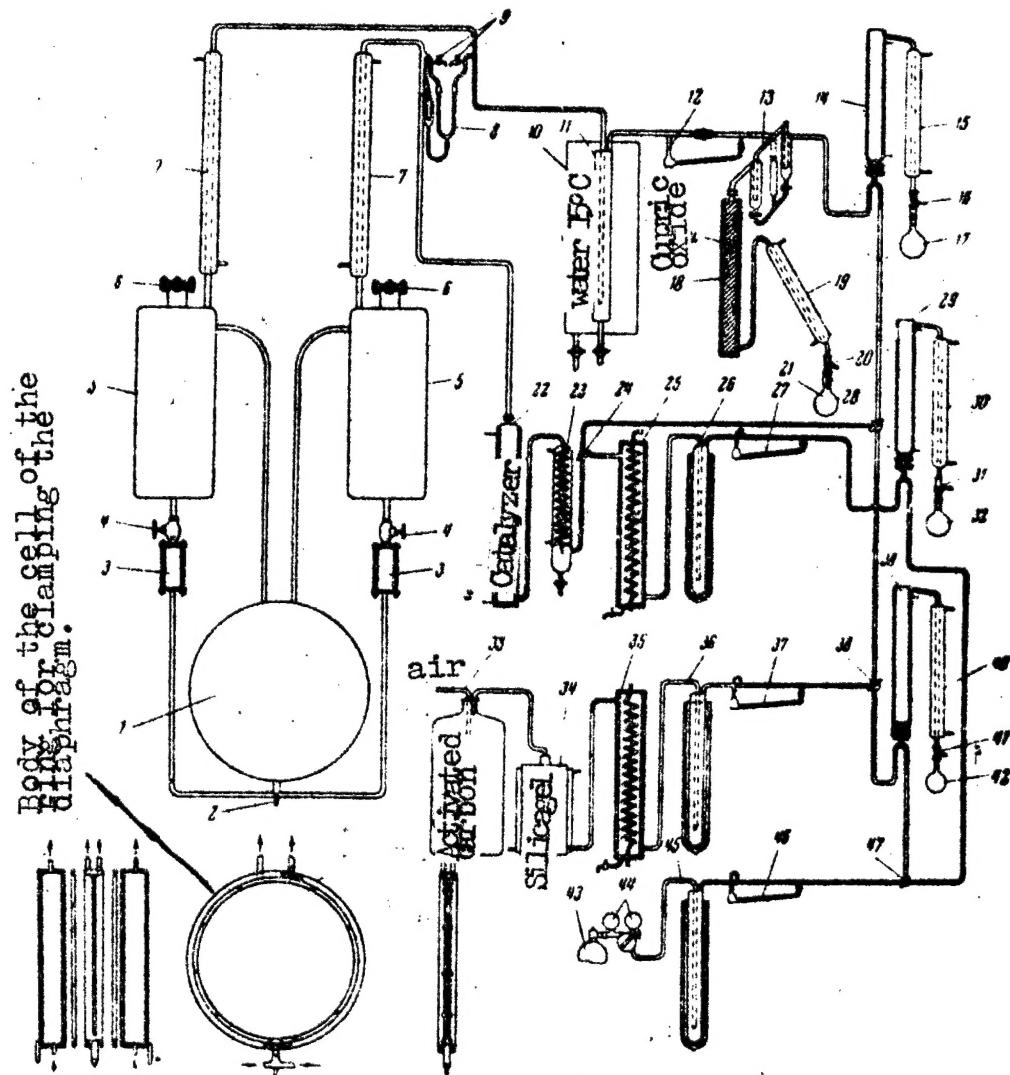


Fig. 1

The blowing through the apparatus and loading of the electrolyte were carried out by means of connecting pipes 6 in the tanks. The connecting pipe is closed by a protective valve of nickel-plated copper foil.

Oxygen and deuterium enter into the coolers 7, made of stainless steel, where the vapors are condensed and splashes are partially retained. Deuterium then enters into the water-cooled splash interceptor 11.

The rate of intake of deuterium is regulated by a calibrated mercury rheometer 12. Deuterium enters through the mercury commutator 13 into the oven 14, which is filled with paladinized pumice and into which electrolytic oxygen or air is introduced. Vapor-like deuterium oxide is condensed in the vertical condenser 15 cooled by running water, and is collected in the flask 17, ground to the cone. It is located at the end of the condenser and its end is below the neck of the flask. The flask communicates with the air by means of the valve of the transition section of part 16.

Gaseous deuterium can be switched over, if desired, for oxidation by the cupric oxide which fills the metal column 18 heated by an electric current. The vapors of deuterium oxide are condensed in the metal cooler 19 and are collected in the ground flask 21. The flask communicates with the atmosphere through the Bunsen valve 20. Cupric oxide is obtained by oxidation of copper by atmospheric oxygen. The switching over of deuterium is carried out by changing the level of mercury in the commutator 13. The whole path followed by deuterium is made of stainless steel. Individual parts are welded together.

Electrolytic oxygen penetrates into the oven 22 filled with paladinized pumice where it is freed from the admixed deuterium. The vapors of deuterium oxide formed in this manner are condensed in the glass cooler 23, from which the condensate is periodically poured off.

Oxygen then enters through the three-way stop cocks 24 and 28 into the oven 14 for burning deuterium; it can also be directed by turning the three-way cock 24 into the system reserved for further drying. Oxygen passes through the metal cylinder 25, in which a copper spiral tube is located. Liquid oxygen passes through this spiral tube, and electrolytical oxygen enters into the controlling quartz trap immersed in a dewar flask filled with liquid oxygen.

The rate of penetration of the electrolytic oxygen is measured with a rheometer 27. The flow of oxygen ends in the oven 29 filled with paladinized pumice. Oxygen from the tank dried by passing through trap 45, and cooled by

liquid oxygen also enters the oven through the three-way stop cock 47. Water vapor, synthetized from electrolytic oxygen and hydrogen from the tank, is condensed in the cooler 30 and the condensate is collected in the flask 32. The lines followed by deuterium and electrolytic oxygen are connected by a U shaped differential mercury manometer 8, which permits the determination of the pressure drop in the system. Electrolytic gases selected for analysis are led out through the stop cocks 9 located over the manometer.

Atmospheric air which enters from the compressor passes through a large layer of activated carbon 33, on top of which are located glass wool and a layer of silica gel cooled by a stream of water 34. The final drying of the air takes place as for oxygen.

Air can be used by turning the three-way stop cocks 38 and 28 so as to burn deuterium in the oven 14 or to burn tank hydrogen in oven 39. This last operation was carried out several times in order to determine the isotope content of tank hydrogen.

Deuterium Oxide Production

Three liters of heavy water (99.9 at .% D) were poured into the electrolyzer before the start of the basic experiment. After addition of K_2O_4 the electrolysis was carried out for 10 hours, after which the electrolyte was poured off and the basic experiment was started.

At the start of the electrolysis the current was 60 amp. It was raised during the course of the experiment to 150 amp. The water samples were selected in quantities of 200-250 g, and each time the first 50 g portion was discarded. When deuterium was burned in the air inside the flask, an increase in the level of the nitrogen resulted, and it therefore was led out through the pipe in section 16. Increase in gas level was also created by burning deuterium in electrolytic oxygen owing to the fact that not all the deuterium was burned.

Thus, contact of deuterium oxide with the atmospheric air was excluded. Immediately after the removal of the sample, the flask was closed with a carefully ground stopper and a ground outer cap. Thus, the purifying of deuterium oxide was started.

The Purification of Deuterium Oxide

It is very important that deuterium oxide be purified and its density measured, so that it will not be diluted with "light" water. The device shown in Fig. 2 serves this purpose.

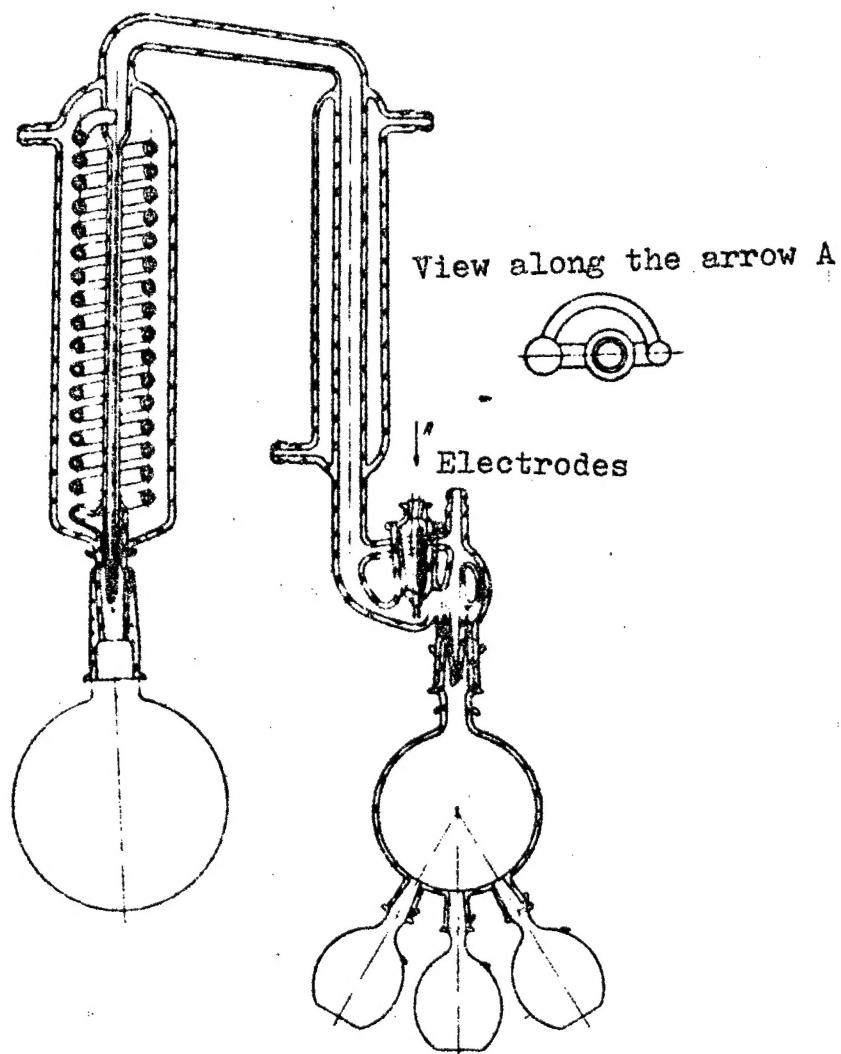


Fig. 2

The flask with the sample was connected to a carefully dried device made of quartz and the product was distilled. The distillate entered into the electrolytic cell and flowed into the receiver. The device communicates with

the atmosphere through a Bunsen valve. When the specific resistance of water exceeded 400,000 ohm, the receiver with water was removed and instead, a reservoir was connected. Three Wirtz' system 11 pycnometers were installed at the bottom of this reservoir. These pycnometers have a capacity of 25 ml and are provided with a neck shaped like a thick-walled capillary having an opening 0.1 mm in diameter.

At the end of the distillation, the reservoir with deuterium oxide was removed from the device and quickly connected to the part used to fill up the pycnometers. The system was alternately evacuated and filled with carefully dried air.

The density was measured as described in 37. Standard water with a specific electrical conductivity of $3 \cdot 10^{-6}$ ohm $^{-1}$, cm $^{-1}$ was used to calibrate the pycnometers. The temperature in the thermostat was maintained within $\pm 0.01^\circ$; it was measured with two calibrated thermometers the readings of which coincided within 0.05° . The weighing was conducted within ± 0.00005 g. The weights were subjected to governmental calibration before and after the experiment. The maximum discrepancy between the data of two calibrations was 0.2 mg for large weights and 0.07 mg for small weights. Each weighing was carried out four, changing the position of the pycnometer and of the weights on the balance each time. The average of weighings was taken and the weights in vacuum were calculated 37.

Let us consider, as an example, the results of weighing one of the pycnometers - empty, and filled with standard water: 8.97993, 8.97995, 8.97996, 8.97994, 8.97993, 8.97997. Average 8.97995.

23.27637, 23.27645, 23.27623, 23.27625, 23.27645, 23.27645, 23.27643, Average 23.27638.

The Results of Measurements

Table 1 and 2 show the average values of measurements of samples of deuterium oxide and samples B taken during electrolysis.

Table 1

Density of samples of deuterium oxide containing electrolytic oxygen

Date	No. of the sample	d_{25}^{25}	d_4^{25}	Date	No. of the sample	d_{25}^{25}	d_4^{25}
18.IV 1950	1A	1,10745,	1,10421,	24.IV 1950	6A	1,10775,	1,10451,
20.IV 1950	2A	1,10754,	1,10433,	25.IV 1950	7A	1,10775,	1,10451,
21.IV 1950	3A	1,10767,	1,10443,	26.IV 1950	8A*	1,10776,	1,10452,
22.IV 1950	4A	1,10772,	1,10448,	27.IV 1950	9A	1,10777,	1,10453,
23.IV 1950	5A	1,10774,	1,10450,	28.IV 1950	10A	1,10778,	1,10454,

* The measurement was made 10 hours after purification of the sample.

Table 2

Density of samples of deuterium oxide containing atmospheric oxygen

Date	No. of the sample	d_{25}^{25}	d_4^{25}	Date	No. of the sample	d_{25}^{25}	d_4^{25}				
Samples obtained by burning deuterium in air											
15.IV 1950	1B	1,10678,	1,10354,	23.IV 1950	8B	1,10766,	1,10442,				
17.IV 1950	2B	1,10727,	1,10403,	24.IV 1950	9B	1,10770,	1,10446,				
18.IV 1950	3B	1,10731,	1,10407,	25.IV 1950	10B	1,10770,	1,10446,				
19.IV 1950	4B	1,10747,	1,10423,	25.IV 1950	11B	1,10788,	1,10444,				
20.IV 1950	5B	1,10755,	1,10431,	26.IV 1950	12B*	1,10760,	1,10438,				
21.IV 1950	6B	1,10765,	1,10441,	Samples obtained by burning deuterium over cupric oxide							
22.IV 1950	7B	0,10788,	1,10444,	26.IV 1950	1B	1,10760,	1,10445,				
				27.IV 1950	2B	1,10770,	1,10446,				

* The neck of the flask burst and the sample was poured into another flask.

After the ninth day of the electrolysis, the density values of samples B remained practically unchanged.

Table 3 gives density values of deuterium oxide, based on the isotopic composition of atmospheric oxygen, which were taken into account while computing its density at 25°.

Table 3
Density of deuterium oxide

Date	No of mea- sure- ments	d_4^{25}	d_2^{25}	Devia- tion from average	Date	No of sam- ples	d_4^{25}	d_2^{25}	Devia- tion from average
24.IV 1950	9B	1,10447 ₁	1,10771 ₁	+0,00001 ₁	26.IV 1950	1B	1,10447 ₀	1,10771 ₀	+0,00001 ₀
		1,10445 ₀	1,10769 ₀	-0,00000 ₀			1,10442 ₄	1,10766 ₄	-0,00003 ₀
		1,10445 ₀	1,10769 ₀	-0,00001 ₀			1,10446 ₈	1,10770 ₈	+0,00000 ₈
25.IV 1950	10B	1,10447 ₈	1,10771 ₃	+0,00001 ₃	27.IV 1950	2B	1,10447 ₂	1,10771 ₂	+0,00001 ₂
		1,10446 ₆	1,10770 ₃	+0,00000 ₃			1,10443 ₄	1,10767 ₇	-0,00002 ₇
		1,10446 ₆	1,10770 ₀	0,00000 ₀			1,10448 ₀	1,10772 ₀	+0,00002 ₀

Average density values: $d_4^{\text{25}} = 1,10446$, $d_2^{\text{25}} = 1,10770$.

Let us compare the densities of samples A and B taken on the same day. The difference in density (Δd) should be related to the excess in the content of O^{18} in heavy water obtained by electrolysis. The difference increased considerably in proportion to the electrolysis of deuterium oxide.

Date	Δd
25.IV	0,00005 ₉
26.IV	0,00006 ₃
27.IV	0,00007 ₅
28.IV	0,00008 ₇

Taking into account that the volume of electrolyte diminished only 2.15 times from 25 to 28 April, it is difficult to explain the observed effect, if we keep in mind the values of the coefficients of separation of isotopes of oxygen given in the literature, during electrolysis. We have no data regarding the coefficients of separation of isotopes of deuterium oxide.

Samples of water obtained by burning tank hydrogen in electrolytic oxygen were subjected to the following methods of purification: oxidation by KMnO_4 in acid and alkali media, and subsequent distillation in a quartz apparatus. After purification by using the floatation method, the density of the samples and of standard water (Δd in γ) were compared. Samples were taken from 19-24 April.

Dates	Δd
19.IV	37
26.IV	61
21.IV	63
22.IV	41
23.IV	37
24.IV	43

Dispersion could be explained by incomplete drying of electrolytic oxygen derived from deuterium oxide and by a partial fractionation of oxygen, because a condensation of oxygen in the traps was noticed in some experiments.

The concentration of heavy isotopes of oxygen in heavy water was measured at the start of the experiment by means of the iron-vapor method [16]. It was found to be equal to 0.08 at.-%¹⁸O.

Results of the three independent determinations (the comparison of densities of samples A and B, the measuring of densities of samples C, and the determination of concentrations of heavy isotopes of oxygen by means of the iron-vapor method) agreed with one another ($60 \pm 20 \gamma$) within experimental error.

Measuring of Density of Deuterium Oxide at 30, 40, and 50°C

The ratios of the volumes of pycnometers V_{30}/V_{25} , V_{40}/V_{25} , V_{50}/V_{25} , were determined by careful calibration. These were equal to 1.00000₀, 0.99999₂, 0.99996₁, respectively. These values differ from those calculated by taking into account the dilation of quartz. The discrepancy could be explained possibly, by the slight evaporation of water from the capillary with increase in temperature. The results of the density measurements of deuterium oxide at 30, 40, and 50° are given in Table 4.

Table 4

Density of deuterium oxide at 30, 40, and 50° C.

№ пр-ти	$t, ^\circ\text{C}$	d_4^t	d_4^t	№ пр-ти	$t, ^\circ\text{C}$	d_4^t	d_4^t
9 B	30	1,09847 ₄	1,10324 ₈	10A	30	1,09855 ₂	1,10322 ₈
		1,09846 ₆	1,10323 ₈			1,09854 ₃	1,10331 ₇
		1,09846 ₂	1,10323 ₈			1,09855 ₉	1,10332 ₁
11 B	40	1,09142 ₇	1,09995 ₈	10A	40	1,09150 ₀	1,10002 ₅
		1,09138 ₈	1,09991 ₈			1,09150 ₈	1,10003 ₈
		1,09142 ₂	1,09995 ₁			1,09154 ₀	1,10004 ₀
13 B ¹	50	1,08264 ₁	1,09571 ₈	10A	50	1,08267 ₁	1,09574 ₈
		1,08261 ₂	1,09568 ₈			1,08268 ₆	1,09575 ₈
		1,08265 ₄	1,09572 ₈			1,08269 ₆	1,09576 ₈

Average values of density of samples d_4^t

$t, ^\circ\text{C}$	30°	40°	50°	$t, ^\circ\text{C}$	30°	40°	50°
Deuterium oxide with atmospheric oxygen	1,10323 ₈	1,09994 ₀	1,09570 ₈	with electrolytic oxygen	1,10322 ₈	1,10003 ₈	1,09575 ₈

Let us establish the density of samples with atmospheric oxygen according to the equation:

$$d_4^t = 1,10446_0 \frac{d_4^t \text{ of the sample}}{d_4^{25} \text{ of the sample}} \text{ (Table 5 line one)}$$

Let us deduct 0.00008₇ (the difference between the density of sample 10A and the average value accepted for deuterium oxide with atmospheric oxygen. See Table 5, line 2, from the density of the sample with electrolytic oxygen.

Table 5

Density of deuterium oxide at 25, 30, 40, and 50°

	25°	30°	40°	50°
d_4^t Average	1,10446 ₀	1,10323 ₈	1,09995 ₈	1,09571 ₈
	1,10446 ₀	1,10323 ₈	1,09994 ₆	1,09567 ₀

Discussion of Results

Let us consider possible errors.

Preparation of deuterium oxide. Incomplete drying of electrolytic oxygen will not reflect on the results, because gas can be moistened by deuterium oxide from the electrolyte in which the concentration of deuterium is practically equal to the concentration of deuterium in the gas when it attains its fixed value. Incomplete drying of the air could cause a dilution of deuterium oxide by "light" water. However as a rule this did not happen because the highest values of the density of samples B coincided with the density of samples B¹, which were prepared by calcining cupric oxide at a high temperature. The penetration of moisture from the air during the synthesis of samples A was excluded. At the same time, the density of samples A and B differed by 0.00002 - 0.00008, and this difference increased regularly, which is explained probably, by the separation of isotopes of oxygen.

Selecting samples of deuterium oxide. A small amount of moisture, which does not exceed 0.0002g (see [37]), remains on the surface of the flask after it has been dried. Therefore, when the volume of the deuterium oxide equals 200 ml the dilution is immeasurably small. There is also no direct relation between the duration of sampling and its density.

No of the sample	Duration of sampling	d_4^{25}
11B	3 h. 25 min.	1.10444 ₂
7B	4 h. 10 min.	1.10444 ₄
8B	5 h. 25 min.	1.10442 ₆

As a rule, the interval between the end of sampling and the beginning of purification did not exceed 0.5 hour.

Purification of samples. Moisture adsorbed on the walls of the apparatus is of no importance if we recall the great volume of the sample. The fact that the device has to be opened twice for a fraction of a minute -- when the receiver is replaced by the reservoir with the pycnometers and after the end of distillation -- is also of no importance.

The filling of the reservoir with deuterium oxide during distillation lasted 1.5 hours. Despite the presence of four necks, the deuterium oxide is not diluted. We became convinced of that after removing the reservoir which contained the pycnometers and which was filled with deuterium

oxide from the device. This was stopped and left for 10 hours. The density of deuterium oxide (sample 8A) was measured after that experiment and was found to correspond to the density of the samples analyzed by the usual method.*

Density measurement! Control tests showed that if pycnometers filled with D_2O are left for 10 hours, their weight does not change appreciably. Usually the pycnometers were weighed directly after filling and thermostating and after a delay of half an hour for weighing.

A detailed analysis of possible errors shows that we had deuterium oxide of a concentration very close to 100%.

Let us evaluate the maximum effect of various errors, which can occur while measuring the density, on the density measurement. 1) errors of weighing, $\Delta d_1 = \pm 0.000006$; 2) errors of converting the weighings to their value in vacuo, $\Delta d_2 = \pm 0.000004$; 3) errors in volume determinations: for the first set of three pycnometers $\Delta d_3 = \pm 0.000008$, for the second set of three pycnometers, $\Delta d_3 = \pm 0.000003$. (The majority of measurements were carried out with the second set of three pycnometers); 4) fluctuations in thermostat temperature, $\Delta d_4 = \pm 0.000004$; 5) error in temperature measurements, $\Delta d_5 = \pm 0.000003$. If these errors were to be added, the error in density measurements would be equal to $0.000025 - 0.000030$. Let us compare the density of deuterium oxide (d_{25}^{25}) obtained by different authors (Table 6).

Table 6

Deuterium oxide density					
Author	Year	d_{25}^{25}	Author	Year	d_{25}^{25}
Taylor, Sel- wood [5]	1934	1.1079	Voskuil, Barash [13]	1945	1.10774
Timmermans [6]	1936	1.10791	Our data	1950	1.10770*
Tronstad, Brun [7]	1938	1.10765*	Vladimirskiy, Kats [15]	1951	1.10770
Johnston [8]	1939	1.10754	Ditto	1952	1.10772
Kirshenbaum [13]	1945	1.10775	"	1953	1.10774

* It is interesting to note that when 100% deuterium oxide was poured from one flask to another (sample 12B) its density diminished by 0.000085. Relative Humidity = 60%.

continued from table 6

* The isotopic composition of the oxygen corresponded to the composition of atmospheric oxygen; in other samples oxygen had the same composition as ordinary water.

The difference in densities due to the variation in concentration of O^{18} is 0.000007. After correction, the average of the last six measurements will not differ from one another by more than 0.00005, and they will be:

$$d_{25}^{25} = 1.10772_5 \pm 0.00002$$

A note by Isborg and Lundberg [17], in which they proposed to introduce a correction into the results of the measurements of Tronstad and Brun appeared in 1954. As you know, the last two scientists obtained two values for the density of deuterium oxide: $d_{20}^{20} = 1.10737$ and 1.10726. The first one is for O^{18} enriched deuterium oxide following the separation of isotopes of oxygen through electrolysis. The second is for samples obtained by burning deuterium in atmospheric oxygen. Isborg and Lundberg consider the first value as more reliable since a penetration of protium is possible when deuterium is burned in atmospheric oxygen. The authors propose to introduce a correction (- 0.00002) into the excessive content of O^{18} (instead of 0.00011, as proposed by Tronstad and Brun). Then, taking into account the coefficient of expansion D_{20} [9], $d_{20}^{20} = 1.10735$ or $d_{25}^{25} = 1.10774$. The density value obtained in this manner corresponds to the data of the most recent studies. However, the correction is apparently too small.

In all studies [9 - 12] dedicated to measuring the temperature coefficient of the density of deuterium oxide the density d_{20}^{20} , according to the data of Tronstad and Brun, was taken as a basis. This data was found to be 0.00006 lower than the values which we obtained. If we introduce the necessary correction, we will obtain the value given in Table 7.

Table 7

Density of deuterium oxide at 30, 40 and 50°

$t, ^\circ\text{C}$	Chang, Chien 10 (1941)	Wirtz 11 1942	Chang Tung 12 1949	Our data 1950	Schrader Wirtz 14 1951	Average
30	1.10327	1.10319	1.10225	1.10324	1.10321	1.10323
40	1.10001	1.09993	1.09998	1.09995	1.09995	1.09996
50	1.09578	1.09563	1.09571	1.09569	1.09568	1.09570

The data which we obtained coincides with the average values of density.

A table of D_2O densities at temperature intervals ranging from 10° to 100° (see 3) was compiled. This table is based on our measurements and on the critical analysis of measurements of the temperature coefficient of expansion of deuterium oxide which existed in the scientific literature up to that time.

In 1954, Isborg and Lundberg [17] compiled a table in which the values coincide within ± 0.00002 with the data in the table compiled by A. I. Shatenshteyn in 1950 [3].

Conclusions

1. An apparatus to obtain deuterium oxide was constructed, and preparations of deuterium oxide of high isotopic purity were made.

2. The density of deuterium oxide was measured at temperatures of 25, 30, 40 and 50°.

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